CHAPTER V

CONCLUSION AND RECOMMENDATION

The Lampang diatomite used as the raw material in this research was composed of 71.09% SiO_2, 11.83% Al_2O_3, 2.86% Fe_2O_3, 0.44% MgO, 0.22% CaO, 1.16% Na_2O, 1.55% K_2O, 4.52% other oxides and 6.35% loss on ignition. It had a microstructure of diatom remains as that of Gas Chrom Q, i.e., Melosira granulata which was a fresh water diatom. It was prepared as the solid support for chromatography by the following steps:

1. A 200 g of the crushed diatomite (1/4 inch diameter) was washed for 3 times with distilled water (the ratio of solid: liquid was 1:5 w/v). Then the coarse minerals which settled in the bottom were separated off by decanting the liquid, clay and diatomite which were separated in the upper part.

2. Clay was removed from diatomite by settling the mixture of clay and diatomite in distilled water, then decanting the supernatant liquid which contained clay off. Sixty per cent of clean diatomite were obtained.

3. A 200 g of the clean diatomite was treated 1 time with 1000 cm^3 10% HCl (the ratio of solid: liquid was 1:5 w/v), the mixture was warmed at 90^o-95^oC for 3 hours and then the diatomite was washed to neutral.

4. The HCl-treated diatomite was flux calcined with 8.55% Na_2CO_3 at 1000^oC for 3 hours. The product obtained was washed with concentrated HCl, then 2.0% KOH solution and finally with distilled water until it was neutral.

5. The flux-calcined diatomite was silanized with 5% v/v

dimethyldichlorosilane in toluene solution.

The composition, physical properties and chromatographic behaviors of this prepared support were studied carefully and compared with those of Gas Chrom Q. The composition of the prepared support was 77.98% SiO_2, 4.07% Al_2O_3, 0.76% Fe_2O_3, 0.24% MgO and 0.22% loss on ignition. The prepared support had lower content of SiO_2 and higher content of Al_2O_3 than Gas Chrom Q did. However, other impurities in the prepared support was clearly lower in content than those in Gas Chrom Q. The prepared support had free fall density of 0.24%, packed column density of 0.26%, hardness of 92.37%, specific gravity of 1.74%, porosity by the wet method of 86.80% and porosity by the dry method of 85.34%. These physical properties was close to those of Gas Chrom Q.

The performance on the chromatographic analysis of non-polar compounds from the prepared support compared to Gas Chrom Q was the same. However, the separation of more polar compounds from the prepared support had two fold better efficiency than that of Gas Chrom Q, even the prepared support had the disadvantage of adsorption due to larger surface area than Gas Chrom Q when 5% liquid phase was used for coating and this results in the requirement of higher loading of liquid phase. Other advantages of the prepared support were the shorter analysis time and the cheaper cost than those of Gas Chrom Q.

Therefore, the prepared support is an excellent solid support for gas chromatography and it was named as "Diatochrom L". The use of Diatochrom L will reduce the imported quantity of solid supports and will lessen the loss of economic balance of country.

In preparation of Diatochrom L, two by-products were obtained. The by product from acid treatment (before calcination) was white, very loose and light. It can be used as a filler for rubber, paper, pigments and others. The by-product from flux calcination was hard so that it

may be used as a filter medium.

Owing to the limitation of time and lack of instruments expecially gas chromatographs which stand at Department of Biochemistry and Faculty of Engineering, the use of these instruments was not convenient. Thus, further study to complete the research should be worthful. The suggested projects are as the followings:

1. To make peak-tailing disappeared by using more per cent loading of liquid phase than this study. The optimum loading can be achieved by varying amount of liquid phase.

2. The application for the analysis of pesticides containing phosphorus should be attempted. This was not carried out in this research since a gas chromatograph with a thermionic specific detector (TSD) sensitive to phosphorus compounds was out of order and it took a long time to maintain.

3. To reduce cost of production, the reagents of industrial grade are suggested to replace those of Analar grade. Owing that the price of crude HCl which is the waste from some local industries in 4 baht/kilogram while the Analar grade HCl is 170 baht/2.5 litres or 58 baht/kilogram. Addition to HCl, soda ash manufactured in Thailand could be used instead of Analar grade Na_2CO_3.

4. By-product from the flux calcination of this study has particle size of -120 mesh (less than 125 μm), indeedly, it is the main-product. Using this product as filter aids should be experimented because the demand of filter aids for factories in Thailand is higher than that of solid support.

5. A flux-calcined diatomite may be used as a catalytic support for some reactors such as the reactor for hydrogenation of vegetable oils. This is an interesting project that should be done because the demand of the catalytic support in Thailand is so high.